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PART I

CERAMIC MATRIX COMPOSITES AS ARMOR MATERIALS

D. Ray Johnson
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The Franklin Institute Research Laboratories

TECHNICAL REPORT AFML-TR-70-54, PART I

April, 1970

AIR FORCE MATERIALS LABORATORY
AIR FORCE SYSTEMS COMMAND
WRIGHT-PATTERSON AIR FORCE BASE, OHIO

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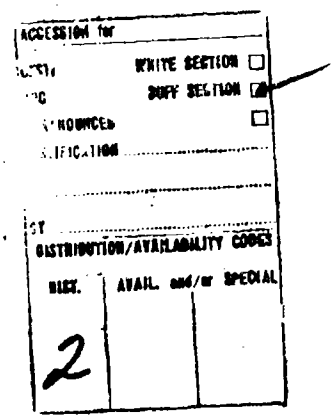

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FOREWORD

This report was prepared by Franklin Institute Research Laboratories under Contract No. F33615-69-C-1659. The contract was initiated under Project 7350, Task 735001, and was administered under the direction of the Air Force Materials Laboratory, Wright-Patterson Air Force Base, Ohio, with John R. Fenter (MAMC) acting as Project Engineer.

This report covers work conducted from 16 June 1969 to 15 January 1970, and was released by the authors in February 1970.

This report has been reviewed and is approved.

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ABSTRACT

Dense ($\geq$ 99%), fine grained ($\leq$ 1-5) samples of titania doped Al_2O_3 and $MgAl_2O_4$ have been fabricated by pressure calcintering. Extremely high values of Young's modulus have been realized in these materials; average values were 66 x 10^6 psi for Al_2O_3, 47 x 10^6 for spinel. Composites were formed by the addition of nichrome wire mesh, single crystal sapphire fiber, and sapphire whiskers. Microstructural examination shows little or no degradation of the wire reinforcement, but the sapphire fibers and whiskers recrystallize and tend to dissolve in the ceramic matrices. Diametral compression tensile strengths are lowered by the inclusion of nichrome wire, but the resultant composites absorb significant amounts of energy during testing after the initial crack. Mechanical properties of sapphire-Al_2O_3/$MgAl_2O_4$ composites were unimpressive with the exception of single crystal fiber - $MgAl_2O_4$ composites. The inclusion of single crystal sapphire fibers in the spinel matrix resulted in a higher ultimate tensile strength than that of the unreinforced spinel ceramic.

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SECTION I

INTRODUCTION

Extremely high hardness and compressive strength are required of materials for ballistic armor. Weight considerations often restrict ballistic materials to those with densities less than 4-5 g/cc. Dense, fine grained polycrystalline ceramics are obviously best suited to these requirements. However, ceramics are characteristically brittle at ambient temperature. Ceramic armor fractures catastrophically under the impact of a projectile and offers little or no "second hit" capability.

Metal wire reinforced ceramics would seem to offer the desirable ceramic properties as well as the toughness required for ballistic applications. Metal wires well bonded in a ceramic matrix should adsorb significant amounts of ballistic impact energy by plastic deformation, holding the ceramic matrix together to prevent catastrophic failure. The higher coefficient of thermal expansion of most metals will leave a ceramic matrix in compression after cooling from forming temperatures. As ceramics normally fracture in tension, a residual compressive stress strengthens the material. This effect is already utilized in tempered glass and prestressed concrete.

Metal wire-ceramic composites are extremely difficult to fabricate by conventional ceramic forming methods. Sintering and hot pressing of the metal oxides, borides, and carbides of interest for ballistic armor application must normally be done at very high temperatures, typically 1500°C and higher; many metals would be molten, or, if the forming were done in air, most would oxidize and degrade badly. While some noble metal alloys are stable in air at elevated temperatures, they do not have the combination of high strength and toughness required for ballistic applications. Furthermore, many ceramic-metal systems are not chemically compatible at elevated temperatures. However, the use of low temperature forming methods and oxidation resistant metals would seem to be a solution to the problems inherent in the fabrication of metal wire-ceramic composites in air.

The approach taken in this investigation is to form composites of nichrome wire with alumina or magnesium aluminate at relatively low temperatures by pressure calcintering. Nichrome is stable in air to ~ 1100°C and possesses good strength and ductility. Alumina and spinel are very hard and strong in compression, making them ideal candidates for ballistic armor. Pressure calcintering, the hot pressing of a decomposing precursor, has been shown to be a feasible method for the low temperature fabrication of small, fully dense, fine grained ceramics.[1-5] At least six groups around the world are currently investigating aspects of this process.

Recent work[6] has shown that very good uniformity and translucency are obtained in pressure calcintered composites of magnesia and stainless

steel wire. More uniform heating within the sample, which decomposes in a strongly endothermic reaction, due to the high thermal conductivity of the metal wires is achieved. These researchers also found that the quality of pressure calcintered magnesia ceramics is very sensitive to starting materials.

A major objective of the present investigation is to develop precursors which pressure calcinter to fully dense, fine grained alumina and magnesium aluminate, and to optimize the chemistry of these starting powders. Further objectives are to demonstrate the feasibility of forming small samples of nichrome wire reinforced alumina and spinel by pressure calcintering, to optimize the forming parameters, and to characterize the mechanical properties of these materials.

The ultimate tensile strength of ceramic materials might also be increased by the incorporation of high modulus filaments such as single crystal sapphire whiskers or fibers. A final objective of the present work is to investigate the feasibility of strengthening pressure calcintered alumina and spinel ceramics by the incorporation of single crystal sapphire whiskers or fibers.

SECTION II

MATERIALS

It is extremely important in low temperature/short time forming operations, as outlined here, that the most intimate mixing possible of reacting components be achieved. This can be done by co-precipitation techniques. However, many such techniques suffer from disadvantages such as adsorption and retention of unwanted and deleterious anionic components, chloride, nitrate, etc., or anionic impurities such as ammonium ions. To avoid this, hydroxides were instead precipitated by hydrolysis with water from alkoxides and the alcohol byproducts evaporated off. Tedious washing and filtering procedures were thus avoided.

1. Alumina

Titania doped alumina ceramics were made from $Al(OH)_3$ + ≈ 2% $Ti(OH)_4$. The $Al(OH)_3$ was obtained commercially* and was doped as follows: A slurry of $Al(OH)_3$ in acetone was blended in a large (5 liters) stainless steel electric blender. The Ti^{+4} was added as a liquid titanium tetraisopropylate,** $Ti[OC_3H_7]_4$, to the slurry of $Al(OH)_3$ in acetone and thoroughly blended. The liquid titanium tetraisopropylate is soluble in acetone and, thus, becomes very intimately mixed into the slurry. The quantity of titanium tetraisopropylate added to the slurry was 8.72%, by weight, of the $Al(OH)_3$; this quantity will theoretically yield 2.38^m/o $Ti(OH)_4$ in $Al(OH)_3$. Distilled water was added dropwise to the blending slurry, 8 moles of water per mole of $Ti[OC_3H_7]_4$, according to the following reaction:

$$Ti[OC_3H_7]_4 + 4H_2O \rightarrow Ti(OH)_4 + 4\ C_3H_7OH \qquad (1)$$

The quantity of water added is twice the amount required to insure completion of the hydrolysis reaction. The slurry was then transferred to a pyrex beaker and the acetone-alcohol-water allowed to evaporate in air. The resulting powder was $Al(OH)_3$ very intimately mixed with $Ti(OH)_4$. During hot pressing, to be described in a later section, the hydroxides decompose to form Al_2O_3 + 4.76^m/o TiO_2. Chemical analyses of C31 $Al(OH)_3$, the $Al(OH)_3$ + $Ti(OH)_4$ powder, and a resulting Al_2O_3 + TiO_2 ceramic sample are given in Tables 1 and 2. A thermogravimetric analysis (TGA) of the powder is given in Figure 1. The TGA was performed*** at a constant heating rate of 5°C/min in an air atmosphere.

2. Spinel

In the early stages of this investigation magnesium aluminate was made with excess magnesia (1 MgO:0.9 Al_2O_3). The excess magnesia is an

*ALCOA C31 hydrated alumina, a product of the Aluminum Company of America, Pittsburgh, Pa.

**Titanium tetraisopropylate, K & K Chemicals, Plainview, N.Y.

***Model 950 thermogravimetric analyzer, E.E. du Pont de Nemours & Co. (Inc.) Wilmington, Delaware.

TABLE 1. EMISSION SPECTROGRAPHIC CHEMICAL ANALYSIS OF ALCOA C-31 $Al(OH)_3$

	Weight Percent of Al_2O_3
SiO_2	0.026
Fe_2O_3	0.003
TiO_2	0.001
Na_2O	0.28
CaO	0.022
Ga_2O_3	0.006
B_2O_3	< 0.001
MnO	0.009
Cr_2O_3	0.0001
MgO	< 0.005
ZnO	< 0.005
CuO	< 0.005
V_2O_5	< 0.001
Cl_2	0.004
Sb	0.01 ppm
SO_3	0.006 ppm
Pb	< 1 ppm
Hg	100 ppm

Table 2. EMISSION SPECTROGRAPHIC CHEMICAL ANALYSIS OF $Ti(OH)_4$ DOPED $Al(OH)_3$ AND RESULTING PRESSURE CALCINTERED TiO_2 DOPED Al_2O_3 CERAMIC

	CONCENTRATION, PPM								
	Ag	B	Cu	Fe	Ga	Mg	Na	Si	Sn
$Ti(OH)_4$ Doped $Al(OH)_3$	2	30	10	45	40	10	0.10%	30	N.D.
TiO_2 Doped Al_2O_3	N.D.	10	10	75	125	100	0.10%	50	50

Limits of Detection for elements not detected (N.D.), PPM Ag(1), As(25), Ba(1), Be(1), Bi(1), Ca(1), Cb(100), Cd(10), Co(10), Cr(1), Ge(5), Hg(50), In(1), Li(25), Mn(1), Mo(100), Ni(10), P(100), Pb(1), Sb(10), Sr(25), Sn(5), Ta(250), Te(25), V(25), W(250), Zn(10), Zr(50)

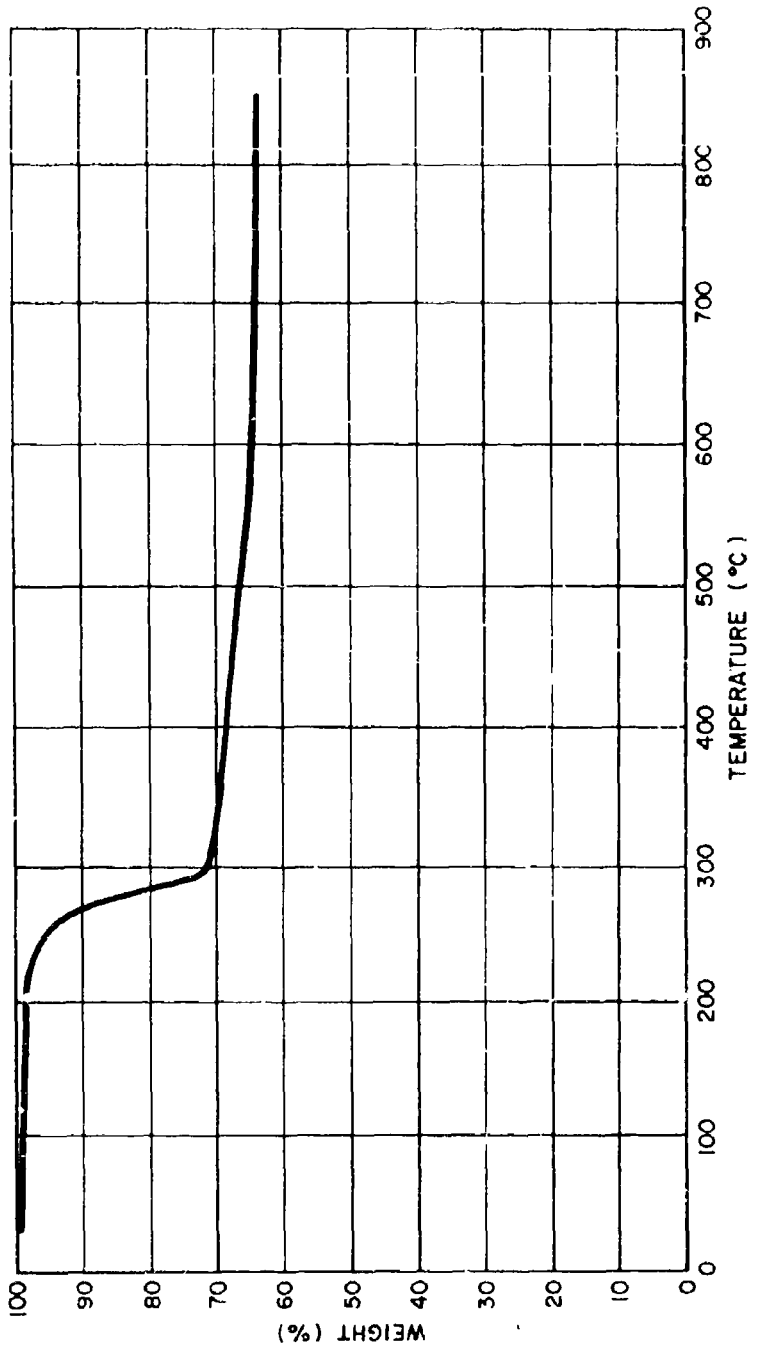


Figure 1. Thermogravimetric Analysis of $Al(OH)_3$ + 2.38 m/o $Ti(OH)_4$

effective grain growth inhibitor. Later, after significant enhancement of Al_2O_3 ceramics had been realized by doping with titania, the authors began making titania doped stoichiometric spinel. The TiO_2 also inhibits grain growth in spinel, and does not have the objection of possible hydration like the MgO.

The procedure for making the nonstoichiometric spinel precursor, $Mg(OH)_2$:0.9 $Al_2(OH)_6$, was as follows: solid aluminum isopropoxide* $Al[OCH(CH_3)_2]_3$ and a solution of magnesium methylate $Mg[OCH_3]_2$ in methanol** were separately ignited to determine the weight fractions of Al_2O_3 and MgO, respectively. Typical results were 26.88^w/o Al_2O_3 in aluminum isopropoxide, 23.6 g MgO per liter of magnesium methylate in methanol. The appropriate quantities of magnesium methylate solution and aluminum isopropoxide to yield MgO: 0.9 Al_2O_3 in the final product were calculated from the gravimetric results. For the typical values given above 199.88 g aluminum isopropoxide are required for a liter of magnesium methylate solution. The aluminum isopropoxide was dissolved in benzene in a 5 liter stainless steel blender, after which the magnesium methylate solution was blended in. Distilled water was added dropwise to the blending solution, 10 moles of water per mole of $Mg[OCH_3]_2$, to hydrolize the alkoxides as follows:

$$Mg[OCH_3]_2 + 2H_2O \rightarrow Mg(OH)_2 + 2CH_3OH \qquad (2)$$

$$Al[OCH(CH_3)_2]_3 + 3H_2O \rightarrow Al(OH)_3 + 3(CH_3)_2CHOH(\text{isopropyl alcohol.}) \qquad (3)$$

$$Mg(OCH_3)2 + 2Al\ [OCH(CH_3)_2]_3 + 8H_2O \rightarrow Mg(OH)_2.2Al(OH)_3 + 2CH_3OH + 6(CH_3)_2CHOH\ (\text{amorphous to X-rays}) \qquad (4)$$

The quantity of water added is twice that theoretically required for complete hydrolysis. As hydrolysis occurs, $Mg(OH)_2$ and $Al(OH)_3$ are coprecipitated in an intimate mixture. The resulting slurry was transferred to a pyrex beaker and the benzene-alcohol-wate· byproducts were allowed to evaporate. The resultant amorphous (mixed hydroxide) powder*** decomposes and reacts to form spinel

$$\underset{(\text{amorphous})}{Mg(OH)_2} + 2\ Al(OH)_3 \rightarrow \underset{(\text{crystalline})}{MgAl_2O_4} + 4H_2O \qquad (5)$$

plus excess MgO during pressure calcintering.

The procedure for making the titania doped stoichiometric spinel precursor is similar to the above with the following exceptions: Typically the quantities of aluminum isopropoxide and magnesium methylate required were 222.02 g aluminum isopropoxide per liter of magnesium methylate solution in methanol. To obtain 2^m/o TiO_2 in the $MgAl_2O_4$, 0.0139 g titanium tetraisopropylate per gram aluminum isopropoxide were required. The titanium

*Practical grade aluminum isopropoxide, Chattanooga Chemicals, Chattanooga, Tenn.

**Magnesium methylate solution in methanol, Morton Chemicals, Chicago, Ill.

***The powder is probably a hydroxide glass and there is no evidence for the separate formation of $Mg(OH)_2$ or $Al(OH)_3$.

tetraisopropylate was added to the blending aluminum isopropoxide-magnesium methylate solution before water was added for hydrolysis. The coprecipitation is the same as above except that $Ti(OH)_4$ is precipitated in the $Mg(OH)_2$ $Al(OH)_3$ glass, which reacts to form spinel + TiO_2 during hot pressing.

The overactive spinel powders produced by the above procedures crack in a "crazy paving" pattern and produce "hour glass" shaped billets during hot pressing. To avoid these difficulties the powder was heated to 350°C overnight to obtain partial decomposition. The 350°C temperature was found empirically; powder heated at that temperature showed uniform shrinkage during hot pressing. The amount of decomposition that has occurred after heating at 350°C can be estimated from the TGA diagram shown in Figure 2. The TGA was performed at a constant heating rate of 0.5°C/min. To achieve higher cold-pressed densities in the spinel precursor, the pre-fired powder was ball milled overnight and passed through a 60 mesh sieve. Powder having been produced and treated by the above procedure still appears amorphous to x-rays.

Typical chemical analyses for the spinel precursor powder and a spinel ceramic sample hot pressed from the powder are given in Table 3. Lattice parameter was measured for each batch of spinel produced. A small amount of the mixed hydroxide powder was calcined overnight at 1000 C and the x-ray diffraction pattern of the resultant spinel determined. X-ray diffraction was measured on a diffractometer* using Ni filtered Cu K_α radiation. The diffraction patterns were determined over the range $2\theta = 10°$ to $2\theta = 150°$. Cell size was calculated from each of the four highest angle peaks. These values were plotted against 2θ and extrapolated to $2\theta = 180°$ to determine the lattice parameter. The measured value was 8.082 Å in each case, in excellent agreement with values reported in the literature.(7)

3. Reinforcing Materials

a. Nichrome Mesh

The nichrome mesh** used in this investigation for nichrome-spinel and nichrome-alumina composites had the following nominal composition: 60 Ni-24 Fe - 16 Cr. The wire was B & S gauge 26 (0.018" diameter) and was woven into a 16 x 16 per inch mesh. The wire mesh was acquired in sheets from which 0.75" disks were cut on a punch press.

b. Single Crystal Sapphire Fibers

A 10' continuous length of 0.010" diameter single crystal sapphire filament was acquired for use in this investigation. The manufacturer*** specifies an average tensile strength greater than 300,000 psi for this material. Short pieces, 1/4" to 3/4", of sapphire filament were hand broken from the continuous length for incorporation into composites.

*XRD-5 X-ray Diffraction Unit, General Electric, Milwaukee, Wisconsin.

**Nichrome Wire Cloth, Newark Wire Cloth Co., Harrison, N.J.

***Continuous single crystal sapphire filament, Tyco Laboratories, Cambridge, Mass.

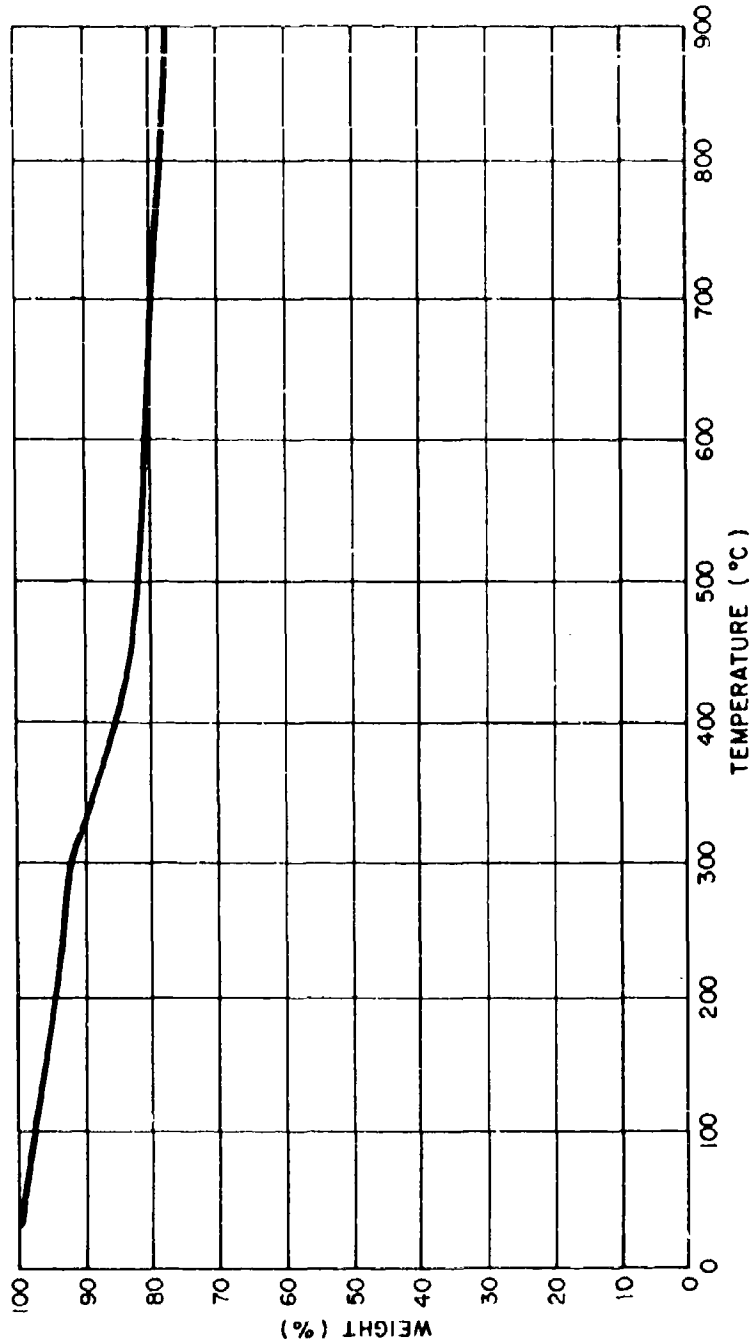


Figure 2. Thermogravimetric Analysis of Coprecipitated $Al(OH)_3$, $Mg(OH)_2$, and $Ti(OH)_4$

Table 3. EMISSION SPECTROGRAPHIC CHEMICAL ANALYSIS OF $Ti(OH)_4$ DOPED SPINEL PRECURSOR AND RESULTING PRESSURE CALCINTERED TiO_2 DOPED $MgAl_2O_4$ CERAMIC

	CONCENTRATION, PPM						
	Ag	B	Cu	Fe	Ga	Ni	Si
$Ti(OH)_4$ Doped Spinal Precursor	N.D.	30	35	75	60	N.D.	60
TiO_2 Doped $MgAl_2O_4$	4	6	50	100	100	125	100

Limits of detection for elements not detected (N.D.), PPM Ag(1), As(25), Ba(1), Be(1), Bi(1), Ca(1), Cb(100), Cd(10), Co(10), Cr(1), Ge(5), Hg(50), In(1), Li(25), Mn(1), Mo(100), Na(25), Ni(10), P(100), Pb(1), Sb(10), Sr(25), Sn(10), Ta(250), Te(25), V(25), W(250), Zn(10), Zr(50)

c. Single Crystal Sapphire Whiskers

The sapphire whiskers* used for composites in this investigation were loose, bulk whiskers, 0.5μ in diameter. The whisker length was 2--20μ. The manufacturer specifies a tensile strength of 3 to 5 x 10^6 psi and a typical purity of 99.5% with minor AlN impurity and traces of Fe and Si. Previous investigators[8] found 0.5μ whiskers more favorable than other sizes for Al_2O_3 composites.

*Type 4-B single crystal sapphire whiskers, General Technologies Co., Reston, Va.

SECTION III

PRESSURE CALCINTERING

A description of the pressure calcintering process and the equipment used in this work is published elsewhere.[1-5] A schematic diagram of the pressure calcintering apparatus is shown in Figure 3. A 50 ton Midvale Heppenstall platen press and a 10,000 psi hydraulic pump were used to apply pressure. The graphite* used for punches and dies is an extruded spectroscopic grade graphite. The material has an apparent porosity of 23%.

A typical time-temperature program and resultant time-density profiles for alumina and spinel are shown in Figure 4. The pressure is applied at room temperature, maintained constant at 4000 psi to 1000°C, and then at 5000 psi from 1000 to 1350°C. These pressures represent the maximum safe values for our porous graphite dies. This has become our standard program for producing small (0.75" dia.), fully dense samples of spinel and alumina.

Spinel and alumina composites reinforced with nichrome mesh were fabricated as follows: nichrome disks 0.75" in diameter were pressed into the die cavity and equally spaced. Any dislodged graphite was blown out of the die. With the lower punch in place the precursor powder was gently vibrated into place between the disks and the upper punch inserted. The arrangement was such as to produce final specimens 0.50 to 0.75" thick with a nichrome disk at each end face and two evenly spaced within.

Single crystal sapphire fibers (0.01" dia.) were incorporated into spinel and alumina samples in a similar fashion. Four layers of fibers were uniformly spaced along the length of each sample, with no fibers at the ends. The layers were "crisscrossed," each one turned 90° from its neighbors. The fibers within a layer were parallel to one another.

Sapphire whiskers were also employed in reinforced spinel and alumina composites. The whiskers (15 wt. %) were mixed with the ceramic precursors by adding them to an acetone slurry in a blender (approximate capacity is one liter). The resulting powder was then hot pressed in the usual fashion.

*UF4S Graphite, Ultra Carbon Corporation, Bay City, Michigan.

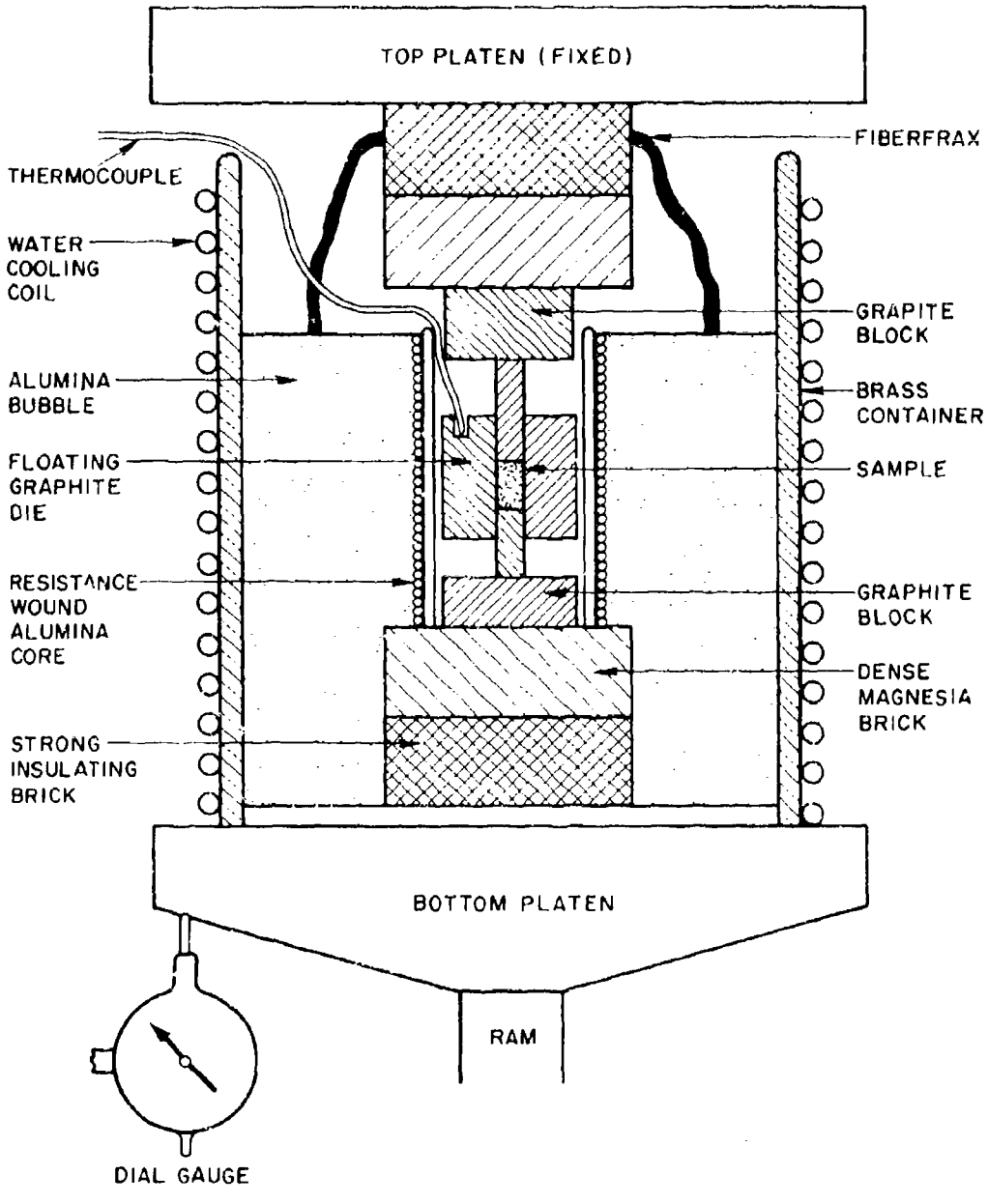


Figure 3. Interior Assembly of Hot Press

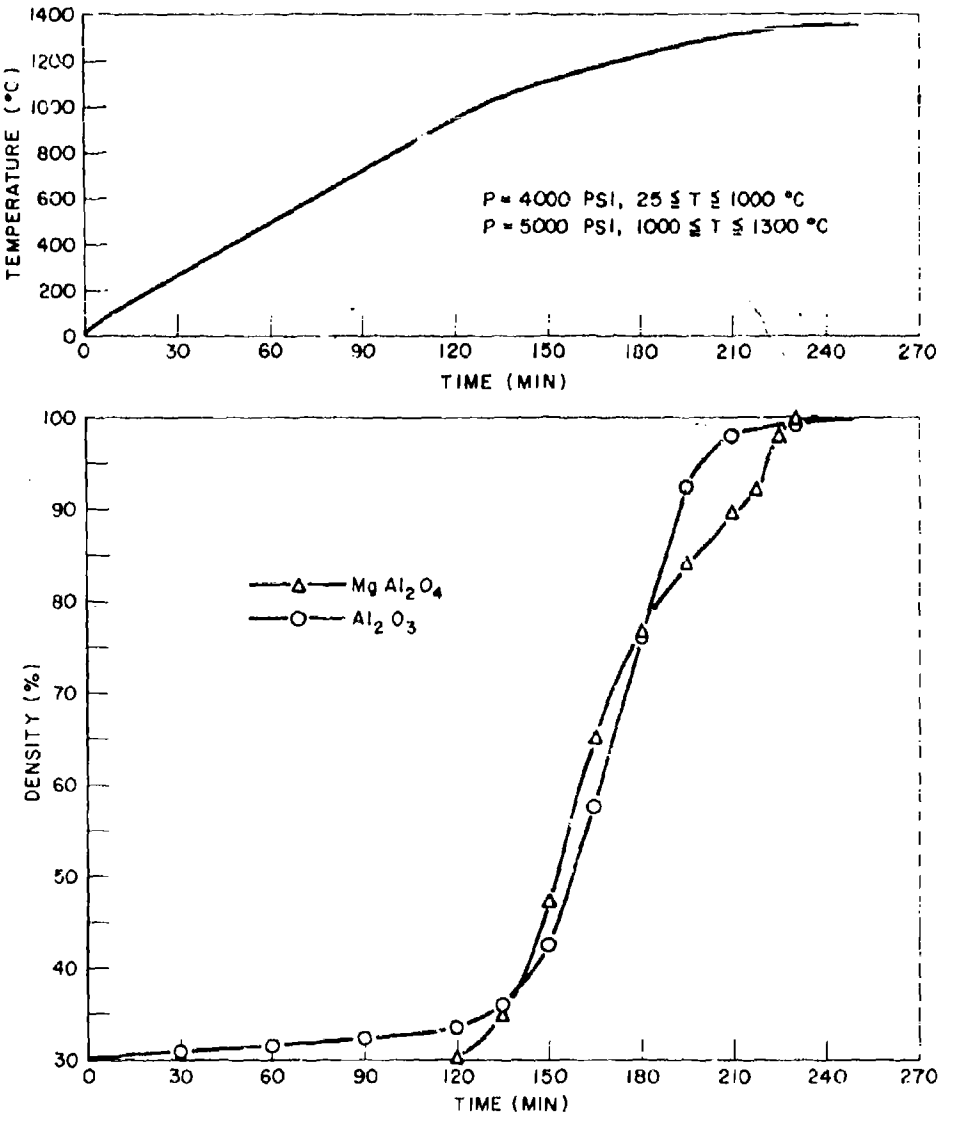


Figure 4. Densification Curves for Pressure Calcintered Alumina and Spinel

SECTION IV

MICROSTRUCTURE

The samples produced by our standard procedures, which were discussed in an earlier section, were typically dense: $\geq$ 99% of theoretical density. A list of the pressure calcintering runs made during this investigation, including all the densities measured, is given in Table 4.

Following earlier work here, the beneficial effect of TiO_2 as a grain growth inhibitor in Al_2O_3 was confirmed. Approximately 4 Mole % TiO_2 in Al_2O_3 was found to be the optimum concentration. The spectacular effect of the TiO_2 dopant on grain size in pressure calcintered Al_2O_3 is apparent in Figure 5, which compares the doped and undoped material.

MgO and TiO_2 were comparable as dopants for $MgAl_2O_4$; both were effective as grain growth inhibitors. The investigators have favored TiO_2 in order to avoid possible hydration effects from the MgO.

Previous work with pressure calcintering[3] indicated that this forming method sometimes results in preferred crystallographic orientation in polycrystalline samples and, naturally, this would affect ballistic properties. The high values of modulus observed in the early stages of the work on alumina, which were measured between the end faces of the cylindrical specimens immediately suggested that "texture" could be responsible. This was found not to be so (see section on modulus also). In order to evaluate the possibility of texture in our pressure calcintered alumina and spinel, diffraction patterns were taken on the faces of samples parallel and perpendicular to the hot-pressing direction. The X-ray diffraction patterns were taken with a diffractometer, using Ni-filtered Cu K_α radiation. Relative intensities were determined by cutting out the peaks on the diffraction pattern with scissors and weighing them on an analytical balance. The results of these experiments are given in Tables 5 and 6. Small differences in diffracted X-ray intensities can be seen at the different sample orientations, indicating possibly a small amount of crystallographic texture. Preliminary efforts to identify the texture using standard pole figure techniques were inconclusive and were therefore discontinued. The preferred orientation in these materials, if any, is subtle and would require a major research effort for complete characterization.

The scanning electron microscope was used to characterize the microstructures of the spinel and alumina ceramics and composites. The X-ray fluorescence capability of the microscope was used to great advantage in determining the extent of reaction between reinforcing wires or fibers and the ceramic matrices.

Average grain size was determined from the scanning electron micrographs by simply measuring the image of a typical grain. Fracture

Table 4. DENSITIES OF PRESSURE CALCINTERED SAMPLES

Sample Number	Material*	Density g/cc	Density, % Theoretical	Comments
503	Al_2O_3 (2^m/o Ti^{+4})	2.28	57.2	
508	Al_2O_3 (undoped)			
523	Al_2O_3 (2^m/o Ti^{+4})			
524	"			Experiment not completed
526	"	3.98	99.9	
531	"	3.61	90.8	BN lined die
532	"	3.75	94.3	1250°C max temp
533	Al_2O_3 (2^m/o Ti^{+4})			1250°C max temp
535	"	3.93	98.6	1300 max temp
536	Al_2O_3 (undoped)			Experiment not completed
537	Al_2O_3 (2^m/o Ti^{+4})			10,000 psi to 900°C
539	Al_2O_3	3.94	99.0	
543	"	3.96	99.4	
548	"			Experiment not completed
552	"	3.07	77.1	
556	"			
558	"	3.88	97.3	1080°C max temp
559	Al_2O_3			
560	"	3.91	97.9	
561	"			1200°C max temp
562	"	3.90	97.8	"
563	"	3.92	98.3	"
564	"	3.97	99.5	"
565	Al_2O_3	3.96	99.3	"
566	"			"
567	"	3.90	97.8	"
568	"	3.77	94.6	"
570	"	3.97	99.6	max temp 1250°C
574	"	3.86	96.7	max temp 1200°C
575	Al_2O_3	3.86	96.8	"
576	"	3.96	99.2	max temp 1300°C
579	"	3.97	99.5	"

*Unless stated otherwise, the level of dopant and hot pressing conditions are those stated in the text.

Table 4. DENSITIES OF PRESSURE CALCINTERED SAMPLES
(continued)

Sample Number	Material*	Density g/cc	Density, % Theoretical	Comments
581	Al_2O_3	3.97	99.6	max temp 1300°C
582	"	3.88	97.2	max temp 1250°C
584	"	3.77	94.5	max temp 1200°C
587	Al_2O_3 ($6^m/o$ Ti^{+4})		97.8	max temp 1250°C
592	Al_2O_3			Experiment not completed
593	Al_2O_3 - Nichrome	3.94		
595	Al_2O_3 ($2^m/o$ Ti^{+4})	3.96	99.2	1250°C max temp
599	Al_2O_3 ($6^m/o$ Ti^{+4})	3.96	99.3	
601	"	3.80	95.4	
604	Al_2O_3	3.94	98.7	
606	Al_2O_3 ($6^m/o$ Ti^{+4})	3.88	97.3	
687	Al_2O_3	3.91	98.0	max temp 1300°C
690	"	3.94	98.8	
761	"	3.82	95.9	
766	"	3.96	99.4	
769	"	3.96	99.3	
771	"	3.94	98.9	
774	Al_2O_3	3.95	99.1	
775	"	3.94	98.8	
778	"	3.95	99.1	
781	"			Experiment not completed
784	"	3.93	98.5	"
788	"	3.97	99.6	
826	Al_2O_3 - Nichrome			
828	Al_2O_3	3.94	98.8	
831	Al_2O_3 - Nichrome			
833	"	3.61		
838	"			
840	Al_2O_3 - Nichrome			
842	"			
845	"			
848				

*Unless stated otherwise, the level of dopant and hot pressing conditions are those stated in the text.

Table 4. DENSITIES OF PRESSURE CALCINTERED SAMPLES

(continued)

Sample Number	Material*	Density g/cc	Density, % Theoretical	Comments
851	Al_2O_3 - sapphire whiskers	3.92	98.2	
853	"	3.79	95.0	
855	"			
858	"	3.82	95.7	
859	"			
862	"	3.00	75.2	
878	"			
886	$MgAl_3O_4$(No Ti^{+4})			Sample cracked
889	"			"
896	"	1.73	48.6	"
899	"	2.61	73.3	"
906	"	2.33	65.4	Sample cracked, 1150°C max temp
907	"	1.25	35.1	"
914	"	2.36	66.2	"
916	"			"
918	Al_2O_3 - Nichrome			
927	$MgAl_2O_4$ (No Ti^{+4})	1.65		1150°C max temp, sample cracked
929	"			"
935	"	3.56	99.9	
940	"	2.58	72.4	1100°C max temp
944	"	2.09	58.7	1100°C max temp
947	"	3.08	86.4	1250°C max temp
951	"	3.32	93.1	"
952				
955	"			
957	$MgAl_{2\ 4}$ - Nichrome			
959	"			
960	"			

*Unless stated otherwise, the level of dopant and hot pressing conditions are those stated in the text.

Table 4. DENSITIES OF PRESSURE CALCINTERED SAMPLES

(continued)

Sample Number		Density g/cc	Density, % Theoretical	Comments
970	$MgAl_2O_4$ (No Ti^{+4})			
975	"			
981	"	3.25	91.2	
990	"	3.20	89.8	
992	"			
1000	$MgAl_2O_4$ - Nichrome	3.49	98.0	
1001	$MgAl_2O_4$			
1006	"	3.55	99.7	
1009	"	3.53	99.0	
1010	$MgAl_2O_4$ - Nichrome	3.52		
1011	"	3.66		
1014	"	3.76		
1018	$MgAl_2O_4$	3.50	98.2	
1020	"	3.52	98.8	
1023	"	3.48	97.7	
1025	$MgAl_2O_4$ - Nichrome			
1027	$MgAl_2O_4$	3.54	99.4	
1028	Al_2O_3	3.95	99.1	
1030	Al_2O_3 - Nichrome			
1034	"			
1036	$MgAl_2O_4$	3.55	99.2	
1038	$MgAl_2O_4$ - Nichrome	3.71		
1039	Al_2O_3 - sapphire fibers			
1045	$MgAl_2O_4$ - sapphire fibers	3.54		
1050	Al_2O_3	3.81	95.6	
1067	$MgAl_2O_4$ - sapphire whiskers	3.37		
1071	Al_2O_3	3.96	99.3	
1080	"	3.78	94.9	

*Unless stated otherwise, the level of dopant and hot pressing conditions are those stated in the text.

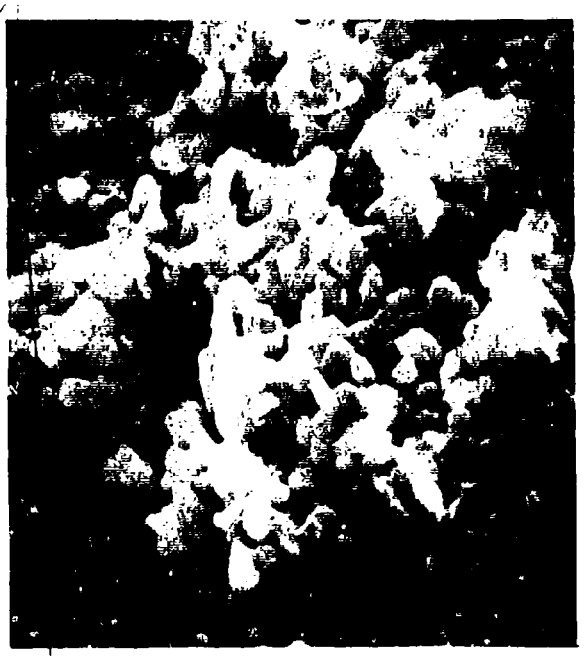
Table 4. DENSITIES OF PRESSURE CALCINTERED SAMPLES

(continued)

Sample Number		Density g/cc	Density, % Theoretical	Comments
1101	$MgAl_2O_4$ - sapphire whiskers	3.38		
1138	"	2.95		
1151	$MgAl_2O_4$ - sapphire fibers	3.55		
1155	Al_2O_3	3.97		
1160	$MgAl_2O_4$ - sapphire fibers	3.55		
1164	"	3.55		
1175	$MgAl_2O_4$ - wire mesh	3.91		
1176	"	3.36		
1177	Al_2O_3 - sapphire fibers			
1191	Al_2O_3	3.95	99.1	
1192	Al_2O_3	3.91	98.0	
1204	$MgAl_2O_4$	3.56	97.5	
1233	$MgAl_2O_4$			

*Unless stated otherwise, the level of dopant and hot pressing conditions are those stated in the text.

(a)



(b)

Figure 5. Comparative Microstructures of Pressure Calcintered Al_2O_3 (a) Undoped, Replica Electron Micrograph, X10,000, (b) 4 m/o TiO_2, Scanning Electron Micrograph, X10,000.

TABLE 5.

X-RAY DIFFRACTION DATA FOR PRESSURE CALCINTERED Al_2O_3

	I/I_o			
hkl	// Pressing Direction	⊥ Pressing Direction	Powder	ASTM Card
012	63	58	47	75
104	128	73	94	90
110	36	45	39	40
113	100	100	100	100
024	50	46	51	45
116	121	77	101	80
018	10	12	10	8
124	36	36	39	30
030	61	65	60	50

TABLE 6.

X-RAY DIFFRACTION DATA FOR PRESSURE CALCINTERED $MgAl_2O_4$

	I/I_o			
hkl	// Pressing Direction	⊥ Pressing Direction	Powder	ASTM Card
111	36	23	24	4*
220	35	30	28	40
311	100	100	100	100
400	59	64	59	58
422	8	28	13	10
511	47	52	50	45
440	71	78	68	58

*This value is in error. The ASTM Joint Committee on Powder Diffraction and N.B.S. indicated that the true value is 44 when questioned about it by the authors. The ASTM Powder Diffraction File is now being corrected.

surfaces were found to be most reliable for examining grain structures. Quantitative metallographic procedures for measuring grain size were not employed, as they would have been invalid for the nonplanar fracture surfaces. Average grain sizes for both the spinel and alumina ceramics were ≈ 1-5μ. The grains were typically equiaxed and the grain size distributions were narrow.

A duplex grain structure was observed in some of the spinel samples. Isolated coarse grained areas were dispersed in the fine grained matrix. A typical area is shown in Figure 6. The linear defect in the micrograph is a scratch. These coarse grained areas are thought to be related to isolated graphite impurities. Occasional optically identifiable bits of graphite that found their way into spinel samples had similar coarse grained areas around them.

Nichrome mesh reinforced alumina and spinel ceramics have been extensively studied by scanning electron microscopy. A typical micrograph of the nichrome-spinel interface is shown in Figure 7. No cracking has been observed at the metal-ceramic interface, even at magnifications up to 10,000X; this was the case for both alumina and spinel. The thermal expansion coefficients of the metal and ceramics differ by a factor of ≈ 2: 17 x 10^{-6} in/in/°C(9) for nichrome, 8.8 x 10^{-6} in/in/°C(10) for alumina, 7.6 x 10^{-6} in/in/°C(10) for spinel. The higher coefficient of expansion for the nichrome should put the ceramic matrix in compression during cooling, thus strengthening the matrix. This is much more desirable than the reverse case in which the wire or fiber has a lower coefficient of expansion, places the matrix in tension, and often results in microcracks at the interface.[6] The micro-probe capability of the microscope was used to determine the extent of reaction between the nichrome wire and spinel. Figures 8a and 8b are plots of relative concentration (intensity of characteristic X-rays emitted) of Ni and Cr, respectively, versus distance across the sample area scanned. Figure 7 shows the area scanned for these results; a line scan was made across the area indicated by the arrows. At magnification as great as 1000X Ni and Cr cannot be detected above background 5μ away from the wire in the spinel. The concentration gradient of these ions is very sharp at the metal-ceramic interface, indicating very little diffusion of Ni and Cr into the matrix. These results suggest very little degradation of the nichrome wire. The results for TiO_2 modified Al_2O_3 were very similar, with the exception that detectable quantities of Cr have diffused 40μ from the wire into the Al_2O_3. The wire reinforcement appears, from these results, to be quite compatible with the fine grained Al_2O_3 and $MgAl_2O_4$ matrices.

No exaggerated grain growth was observed in the matrix materials near the wire; the grain structure at the interface appeared unaltered relative to the bulk. This is apparent in Figures 6 and 9, scanning electron micrographs of wire reinforced spinel and alumina, respectively. These observations are consistent with the scanning electron microprobe results: no appreciable counter diffusion of ions occurred at the interface.

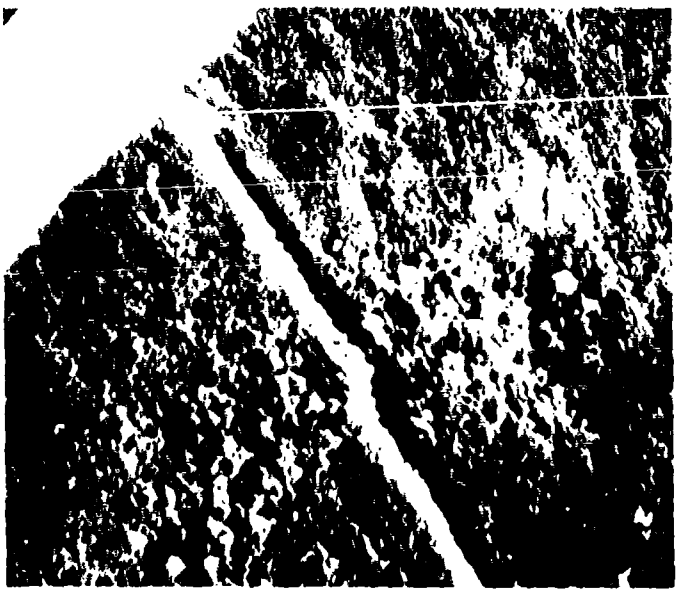


Figure 6. Scanning Electron Micrograph of Nichrome Wire Reinforced Spinel Showing Fine Grained Structure Adjacent to the Wire and Isolated Coarse Grained Area X2,000
(The Linear Defect is a Scratch)

Figure 7. Scanning Electron Micrograph of Spinel-Nichrome Wire Interface. Arrows Indicate Line Scanned for X-ray Flurescence. X1,000

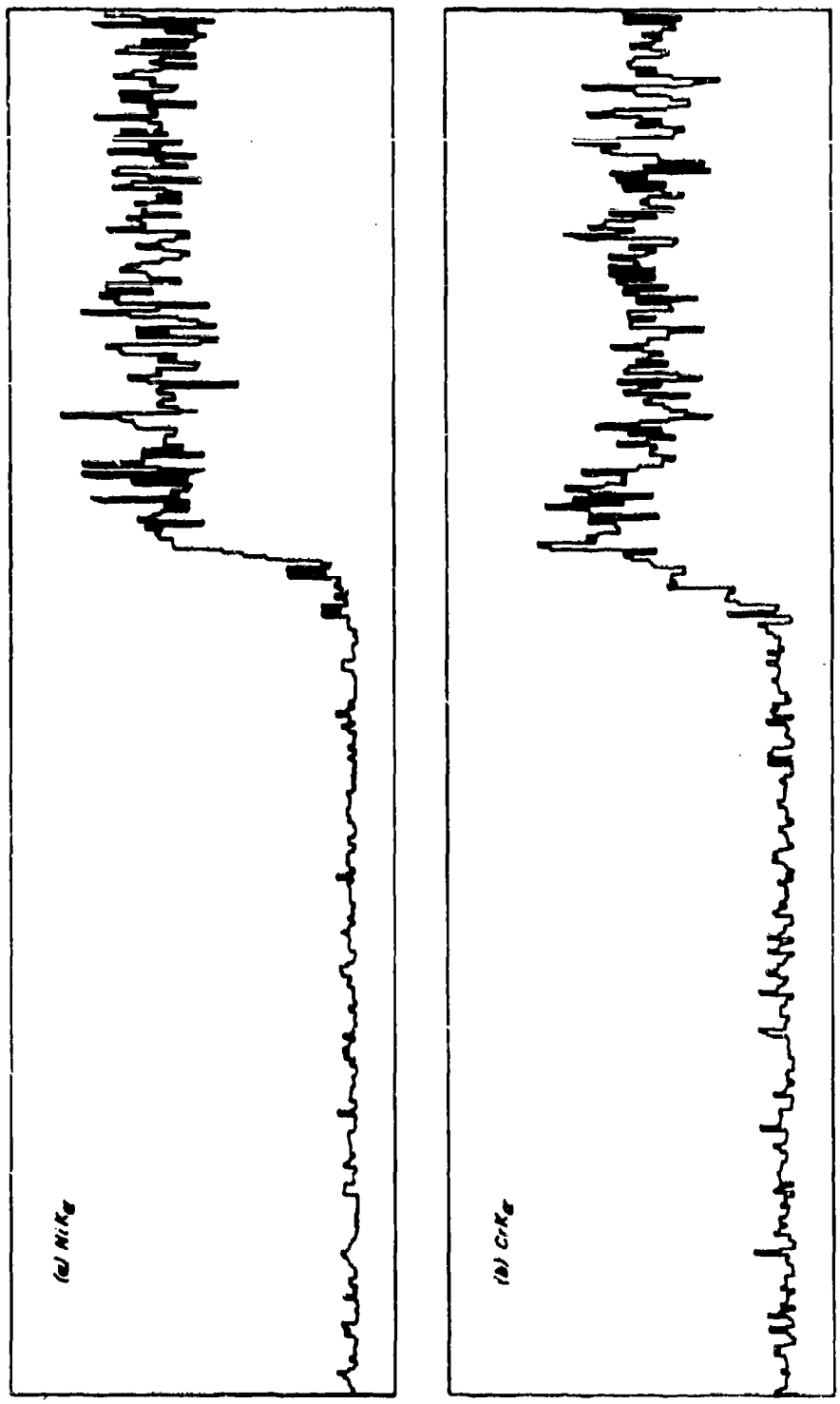
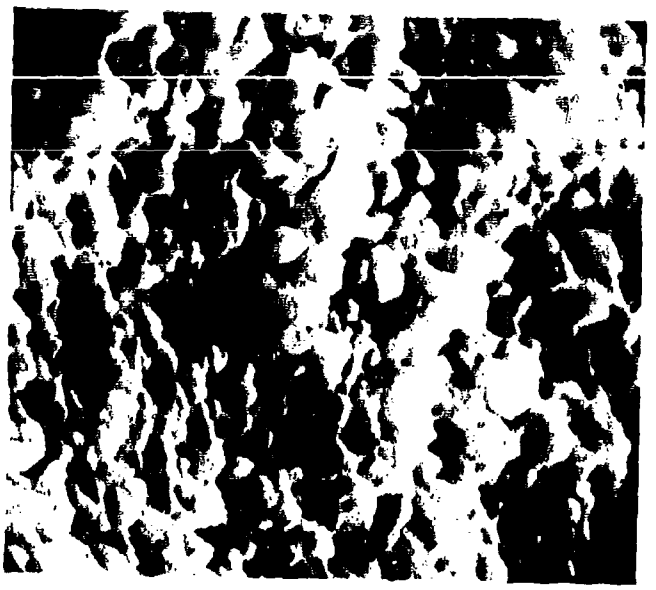
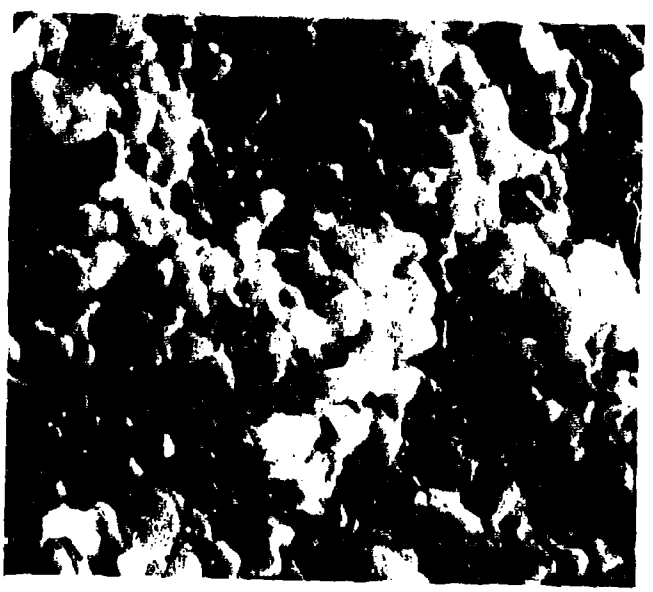


Figure 8. Intensity of Characteristic X-rays Versus Distance Across the Nichrome Wire-Spinel Matrix Interface, a) Ni K_α, b) Cr K_α



(a)



(b)

Figure 9. Scanning Electron Micrograph of Nichrome Wire Reinforced Alumina Showing the Grain Structure (a) Immediately Adjacent to the Wire and (b) Removed From the Wire. Fracture Surface X10,000

Sapphire whiskers could not be found in the microstructures of dense, pressure calcintered spinel and alumina composites fabricated with 15% by weight whiskers. Instead, porous, coarse grained areas were uniformly distributed throughout the material. Figures 10 to 12 illustrate these areas in spinel composites. X-ray fluorescence results from the scanning electron microscope show that these areas are alumina rich. Figure 13 shows the intensity of characteristic Al K_α X-rays emitted while scanning across the area shown in Figure 11; the position of the line scan is indicated by arrows. Apparently the whiskers have gone into solution, leaving highly porous, large grained, Al_2O_3-rich areas in the spinel microstructure. The dissolution of sapphire whiskers in oxide materials has been previously observed in conventional hot pressing[8]. The decomposing precursors in a pressure calcintering system are extremely reactive, making dissolution even more likely than in the case of normal hot pressing. The porous, coarse grained areas left behind by the whiskers would seem to be preferred sites for crack initiation. One would not expect these composites to perform well in ballistic tests.

Visual inspection of spinel samples containing single crystal sapphire fibers shows that many of the fibers break into small fragments during fabrication. Microstructural examination shows that these pieces are polycrystalline, with average grain size $\approx$ 1μ. Each fragment is surrounded by a ring of coarser material: average grain size $\approx$ 3-5μ. Figure 14 illustrates a typical sapphire fragment. The single crystals apparently recrystallize during pressure calcintering.

Figure 10. Macrograph of Pressure Calcintered Spinel Containing 15% by Weight of 0.5μ Sapphire Whiskers. Fracture Surface, X5.7

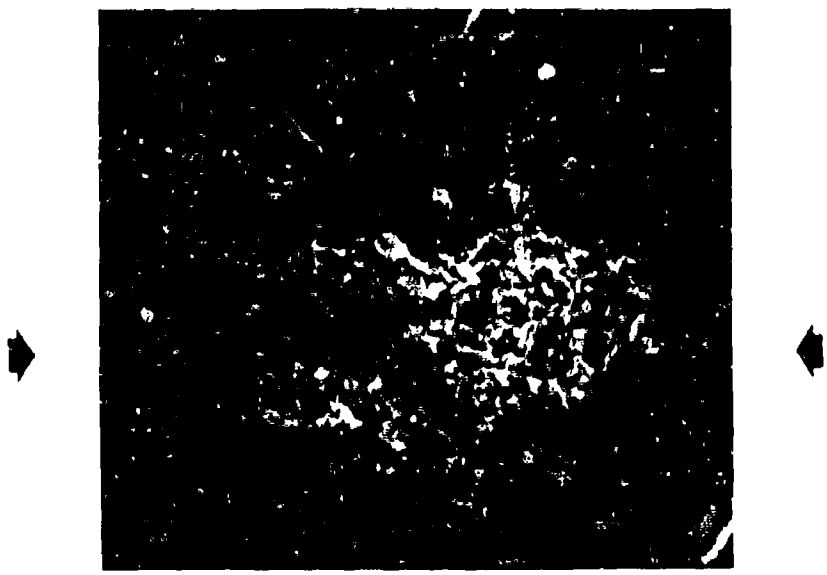


Figure 11. Scanning Electron Micrograph of Pressure Calcintered Spinel Containing 15% by Weight 0.5μ Sapphire Whiskers. Arrows Indicate Line Scanned for X-ray Fluorescence. X600

Figure 12. Scanning Electron Micrograph of Pressure Calcintered Spinel Containing 15% by Weight 0.5μ Sapphire Whiskers. X3,000

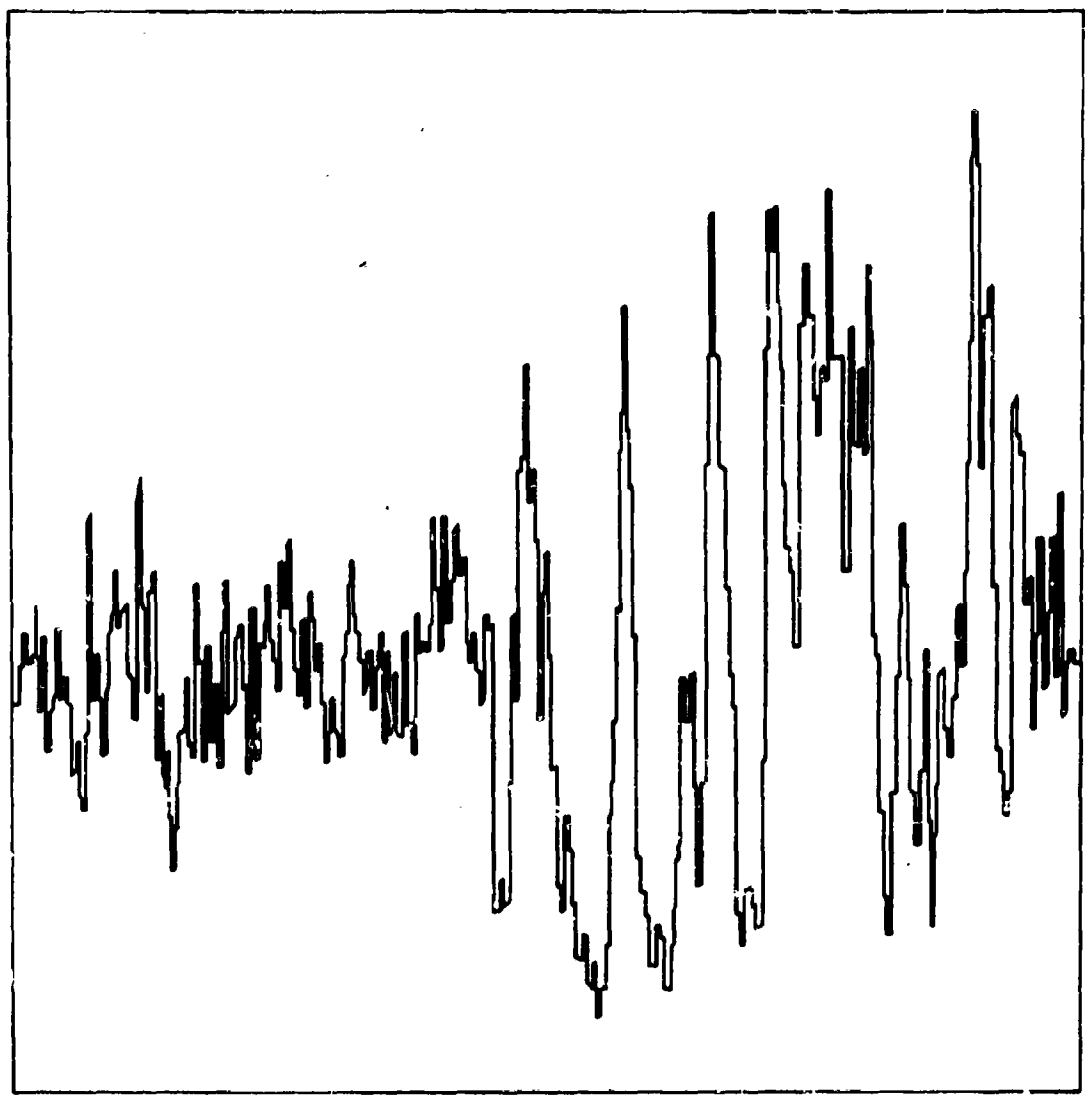
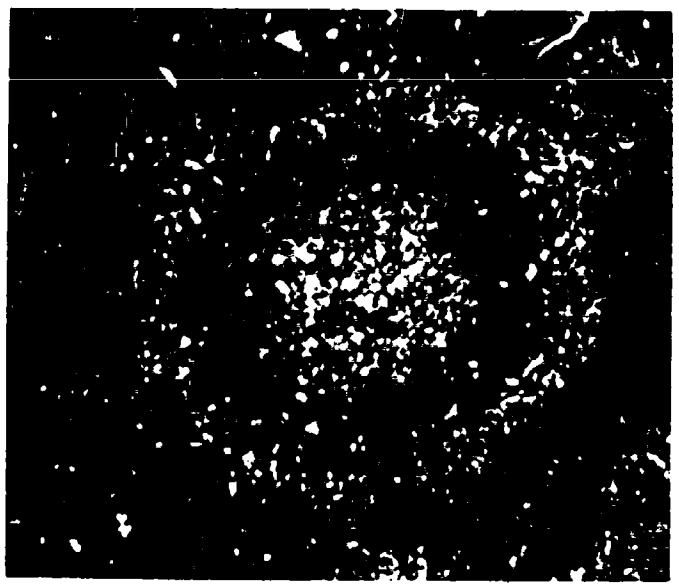
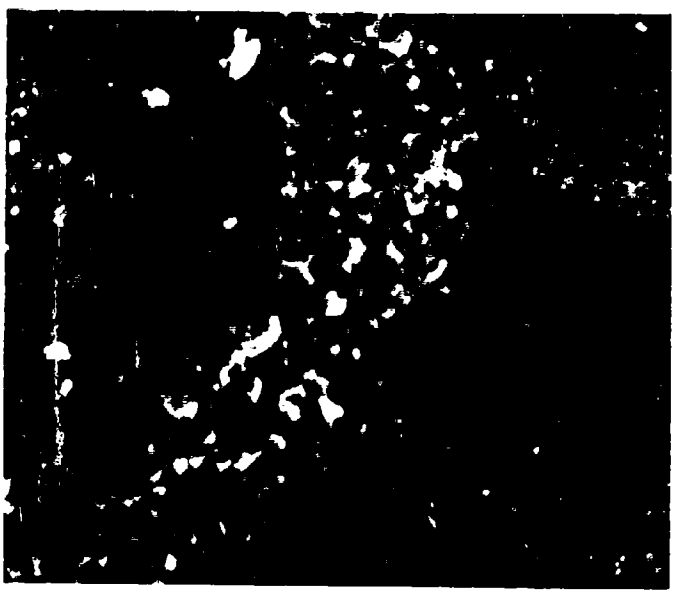


Figure 13. Intensity of Characteristic Al K_α X-rays Versus Distance Across the Sample Area Scanned. Refer to Fig. 11



(a)



(b)

Figure 14. Scanning Electron Micrograph of Sapphire Fiber Reinforced Spinel. (a) Sapphire Fragment and Surrounding Coarse Grained Ring. X300 (b) Coarse Grained Ring. X1,000

SECTION V

MECHANICAL PROPERTIES

1. Tensile Strength

Tensile strengths were measured by the diametral compression method. In this test a right circular cylindrical specimen is compressed diametrically between two flat platens. The maximum tensile stresses are developed normal to the loading direction across the loaded diameter. These tensile stresses cause the cylinder to fracture along the diametral plane joining the lines of contact of the specimen and the platens. Since the tensile stresses are directly proportional to the applied load, the tensile strength can be computed from the load at fracture:

$$\sigma_m = \frac{2P}{\pi dt}$$

where

P = applied load

d = specimen diameter

t = specimen thickness

σ_m = maximum tensile stress

Spriggs, *et al*[11] noted that the diametral compression tensile strengths of a large number of ceramic materials were approximately one half the transverse bending strengths.

A sample holder similar to that described by Spriggs, *et al*[11] was used for the diametral compression tests. A schematic diagram of the sample holder is shown in Figure 15. The samples were tested "as pressed" with no surface treatment other than a rough surface grinding. An Instron testing machine was used to load the samples to failure at a strain rate of 0.002 in./min.

The tensile strengths of the samples tested in this investigation are listed in Table 7. The measured values have been multiplied by 2.0 to make them comparable to frequently cited transverse bending strengths.

Typical "optimum" values for mechanical properties of dense, fine grained polycrystalline alumina and spinel have been compiled by Lynch, *et al.*[12] Their values for the bend strength of alumina and spinel are as follows: Al_2O_3 - 62-68000 psi, $MgAl_2O_4$ - 32,000 psi. These values are somewhat higher than the values derived from diametral compression tests in this investigation. But, of course, great care is required to achieve optimal values and the lower strength values here are thought to be the result of the rough method. To further resolve the tensile strength of these materials, 4.0" rounds of FIRL alumina and spinel ceramics and composites will be supplied to our sponsor to be tested in uniaxial tension.

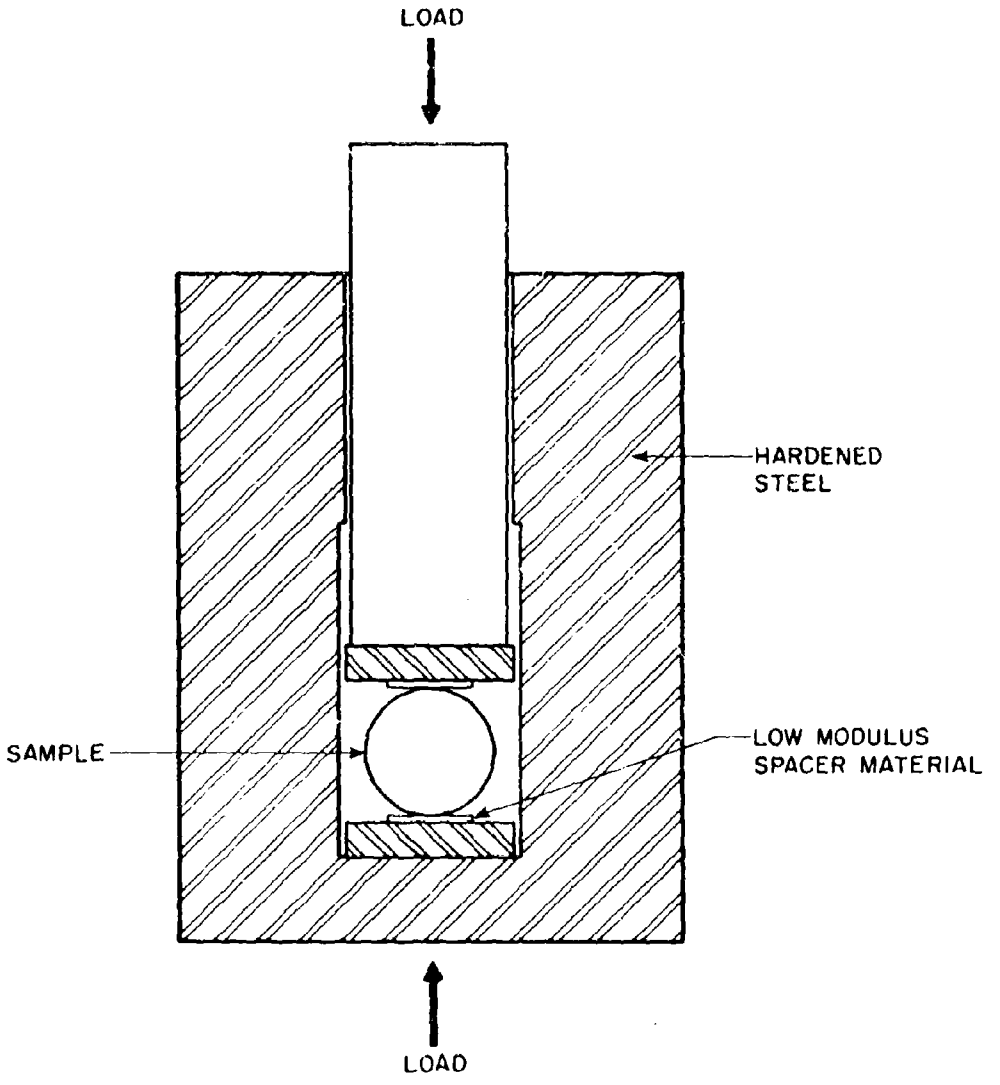


Figure 15. Sample Holder for Diametral Compression Strength Tests

Table 7. DIAMETRAL COMPRESSION TENSILE STRENGTHS

Material	Sample Number	σ_m,psi	Density g/cc	Density % Theoretical
Al_2O_3	769	>53,126*	3.96	99.3
"	766	38,318	3.96	99.4
"	761	39,826	3.82	95.9
"	788	47,198	3.97	99.6
"	774	20,968	3.95	99.1
"	778	44,870	3.95	99.1
"	828	46,284	3.94	98.8
	Average	41,512		
Al_2O_3 - Nichrome	831	27,406		
"	826	30,352		
"	833	9,672	3.61	
"	840	23,756		
"	842	27,026		
"	845	18,760		
Al_2O_3 - Nichrome	848	21,122		
"	838	15,210		
	Average	21,663		
Al_2O_3 - Sapphire Fiber	1177	23,038		
Al_2O_3 - Sapphire Whiskers	851	30,624	3.92	98.2
	853	37,660	3.79	95.0
	Average	34,142		

*This sample did not fracture under the highest load attainable on our Instron testing machine.

Table 7. DIAMETRAL COMPRESSION TENSILE STRENGTHS
(continued)

Material	Sample Number	σ_m, psi	g/cc	Density % Theoretical
$MgAl_2O_4$ (MgO Rich)	935	11,302	3.56	99.9
"	947	24,350	3.08	86.4
"	951	23,776	3.32	93.1
"	992	20,064	3.49	98.0
$MgAl_2O_4$	1006	27,008	3.55	99.7
"	1009	24,486	3.53	99.0
"	1023	13,030	3.48	97.7
"	1027	28,120	3.54	99.4
	Average	21,515		
$MgAl_2O_4$ - Nichrome	1010	17,300	3.52	
"	1011	10,950	3.66	
"	1014	7,680	3.76	
"	1038	8,810	3.71	
"	1175	14,588	3.91	
	Average	11,865		
$MgAl_2O_4$ - Sapphire Fiber	1160	21,348	3.55	
"	1164	22,040	3.55	
"	1151	27,906	3.55	
	Average	23,765		

Ballistic failure is exceedingly complex involving tensile and shear failure modes and will be closely related to "toughness," a parameter exceedingly little understood in the ceramic art. In the absence of a good appreciation of the various factors that may be important it is questionable whether refined mechanical testing is worthwhile; rather, ballistic tests themselves must be rapidly conducted for evaluation.

The strength data for the nichrome mesh-ceramic matrix systems are very interesting in light of the intended ballistic application for these materials. The nichrome reinforced composites fractured at a lower stress than the unreinforced matrix material. However, these composites continued to support an increasingly load after the initial fracture. Figure 16 shows representative load-deformation curves for Al_2O_3 and an Al_2O_3 - nichrome composite. The loads are normalized for sample lengths to make the curves for the two samples comparable. The unreinforced Al_2O_3 ceramic failed catastrophically. The nichrome reinforced Al_2O_3 composite showed only the main central crack after initial failure, with the stored elastic energy absorbed, presumably, by plastic deformation of the wire. Figure 17 illustrates the dramatic difference between the types of fracture encountered. If, after the initial diametral crack, loading is continued on the Al_2O_3 - nichrome composite, the sample will continue to support an increasing load with approximately the same load-deformation curve as before fracture. Finally, cracking, of the matrix, delamination, and deformation of the wire occur as the sample gradually crushes.

The spinel-nichrome samples also continued to support an increasing load after initial fracture. However, some of the spinel-nichrome composites initially failed by delamination. Other wire configurations may be better for the spinel. Delamination of the wire from the spinel matrix might absorb an appreciably quantity of energy in ballistic impact; further judgment will be reserved until ballistic testing is completed.

These results cannot, of course, be directly converted to a ballistic evaluation, but it seems apparent that the nichrome reinforced composites can absorb considerably more energy in failure than can the unreinforced oxides.

The strength data for the whisker reinforced composites are not very impressive. The whiskers do not reinforce the matrix; they, instead, weaken the material. This is no doubt due to the dissolution of the whiskers during fabrication and the accompanying porous coarse grained areas in the microstructures. The sapphire fiber-alumina composites are likewise unimpressive. However, single crystal sapphire fibers do apparently strengthen the spinel ceramics. Limited mechanical data for this system indicate that the fiber reinforced spinel is stronger than the unreinforced matrix, in spite of the fragmentation and recrystallization of the sapphire fibers during fabrication of the composites.

2. Elastic Modulus

Measurements of elastic modulus were performed on a Sperry Rand UM715 ultrasound reflectoscope, using the reflection method. The transit

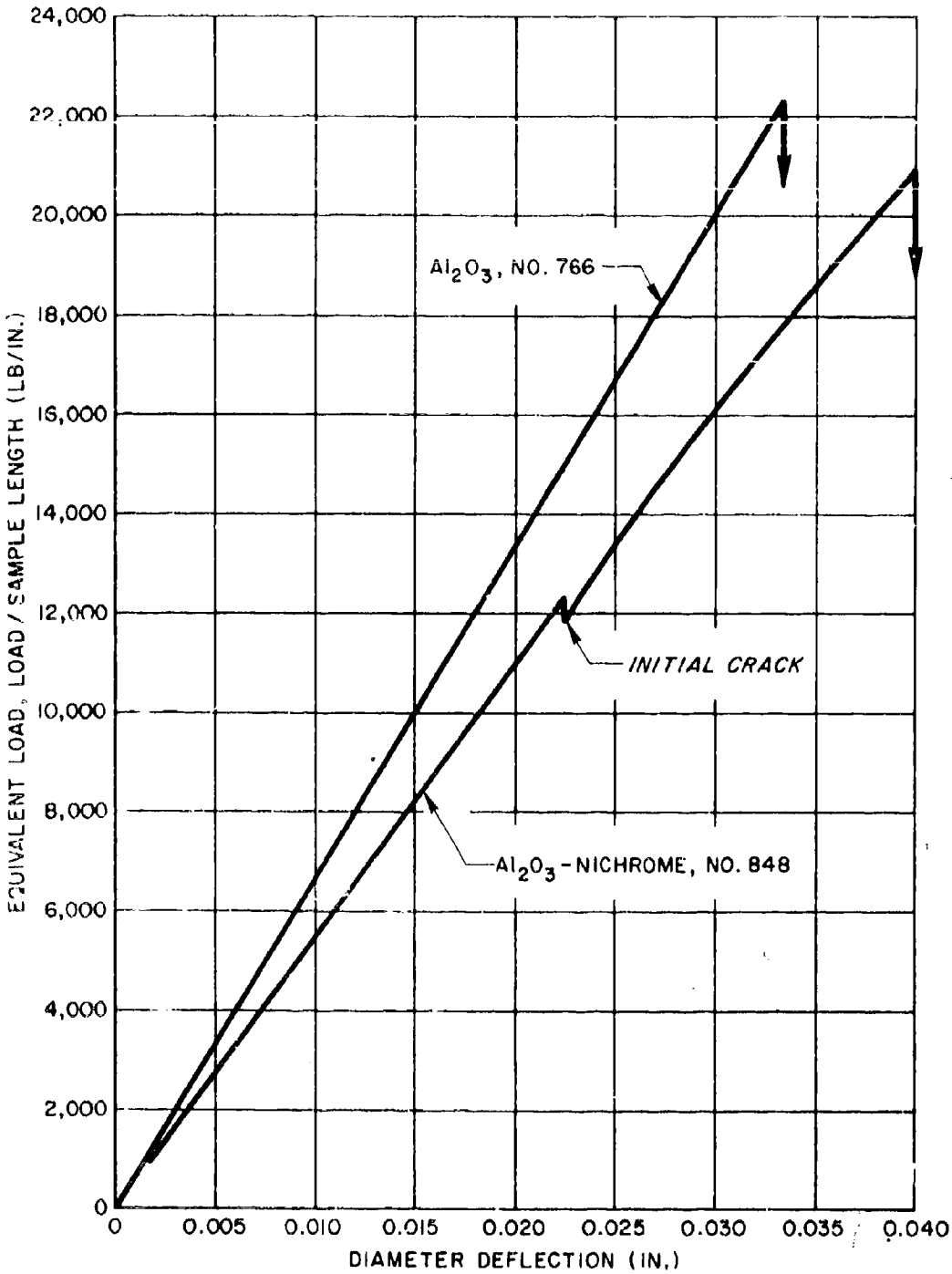


Figure 16. Load-Deformation Curves for Alumina Ceramic and Alumina-Nichrome Wire Mesh Composite

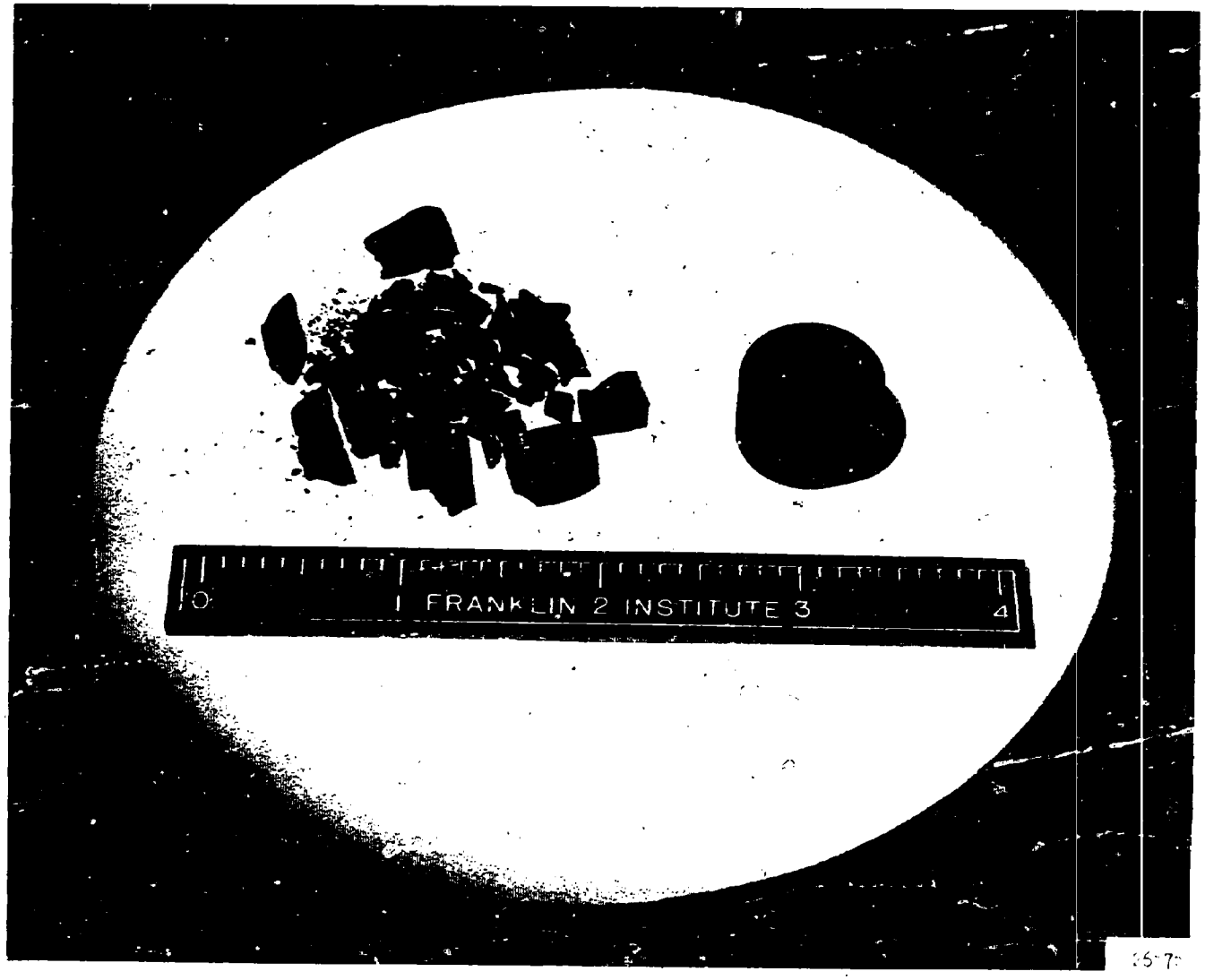


Figure 17. Diametral Compression Test Samples of Alumina (Left) and Nichrome-Alumina Composite (Right) Showing Type of Fracture

tim resolution for ultrasound pulses on the reflectoscope is $\gtrsim$ 1 μ sec. Since the typical transit time of the samples is of the order of several μ sec, we have considerably improved the resolution by taking the output from the reflectoscope to a tektronix oscilloscope. The time scale on the oscilloscope is 0.01μs enabling the measurement to be made to an accuracy of 1%.

In this method an ultrasound transducer was attached to one face of the cylindrical sample. The faces of the samples had been ground to a smooth finish on a belt sander. The time required for a sound wave to travel through the sample and bounce from the opposite side back to the transducer was read on the time calibrated scale of the oscilloscope. The thickness of the samples was measured with a micrometer. The velocity of sound travel through the material is obtained from the relation $2x/t$, where x = sample thickness, t = time. The elastic modulus E is found from the relationship $V = (\frac{E}{\rho})^{1/2}$ where V is the velocity of ultrasound propagation and ρ, the sample density. The measurements were usually made at 2.55 or 10.00 m c/s.

The modulus of a Tungsten sample was measured in order to check the calibration of the apparatus. The Tungsten sample had been arc cast and extruded by the manufacturer.* The material was 100% dense and contained less than 20 ppm carbon and less than 35 ppm oxygen. The measured value for the modulus of tungsten was 59.8×10^6 psi, which is in excellent agreement with the literature value,[13] 59×10^6 psi.

The elastic modulus data from this investigation are listed in Table 8. The modulus of elasticity values for the FIRL pressure calcintered, titania doped alumina and spinel are extremely high. Typical optimum values listed by Lynch, *et al*[12] are 59×10^6 psi for Al_2O_3 and 34×10^6 psi for $MgAl_2O_4$. Good values of moduli as a function of crystallographic axes in single crystal alumina[14], done at N.B.S. are also lower than the results found here; the FIRL materials have the highest apparent values of Young's modulus known to these investigators. The extremely high modulus of these materials may be very significant in their ballistic performance. A brief study of the variation of modulus with the direction of measurement, i.e., parallel and perpendicular to the pressing direction supported the X-ray data that little or no "texture" exists.

Modulus measurements made parallel and perpendicular to the pressing direction yielded the following data:

Sample No.	Material	Elastic Modulus, psi x 10^6 // Passing Direction	⊥ Pressing Direction
579	Al_2O_3	64.6	65.5
1039	Al_2O_3 + sapphire fiber	65.6	70.2
1045	$MgAl_2O_4$ + sapphire fiber	47.4	42.1

*Atlantic Research Corporation, Alexandria, Virginia.

TABLE 8. ELASTIC MODULUS DATA

Material	Sample Number	Thickness cm	Transit Time, μ sec.	Density, g/cc	Elastic Modulus, psi x 10^6
Al_2O_3	774	0.870	1.60	3.95	67.5
"	778	0.890	1.70	3.95	63.3
"	788	0.812	1.55	3.97	63.3
"	828	0.900	1.70	3.94	64.7
"	769	0.814	1.50	3.96	67.4
"	784	0.864	1.75	3.93	56.2
"	576	0.733	1.30	3.96	73.3
"	570	1.222	2.35	3.97	62.6
"	1028	1.030	1.85	3.95	71.0
"	579	0.875	1.60	3.97	69.0
				Average	65.8
Al_2O_3 - Sapphire Whiskers	853	0.770	1.55	3.79	53.2
"	858	1.548	3.20	3.82	48.4
"	862	1.140	3.00	3.00	25.2
				Average	42.4
$MgAl_2O_4$	992	0.839	1.70	3.49	49.6
"	1006	0.500	1.01	3.55	51.7
"	1009	0.576	1.10	3.53	51.5
"	1020	0.572	1.30	3.52	39.7
"	1018	0.326	0.70	3.50	44.0
"	1023	0.620	1.30	3.48	46.0
"	1027	0.581	1.20	3.54	48.3
				Average	47.3
$MgAl_2O_4$ - Sapphire Fiber	1151	0.618	1.30	3.55	37.1
"	1164	0.665	1.40	3.55	46.0
				Average	41.6

The values for sapphire fiber composites reflect only the modulus of the matrix material, as no sonic reflections from the sapphire were observed. The differences in modulus with direction of measurement are insignificant for the alumina ceramic. The apparent differences for the sapphire fiber composites may be important, and will be further investigated.

Young's modulus could not be determined for the nichrome reinforced alumina and spinel composites by the sonic method because the sound waves were scattered in all directions by the wire mesh; an important finding for the ballistic performance of these composites if the shock wave from ballistic impact were likewise scattered.

3. Microhardness

Microhardness values were determined with a Wilson Tukon microhardness tester, using a Knoop indentor and a 1000g load. The condition of the indentor was checked by making indentations in a standard calibrated test block, which had been indented by the Wilson Company. The test block had a Knoop hardness of 755 Kg/mm^2 and had been indented under a 1000g load by the manufacturer. Indentations in the standard test block made by our instrument (1000g load) were virtually identical to the standard indentations.

The filar eyepiece and objective lens in the optical system of the tester were calibrated against a standard magnification scale from an optical microscope. All the measurements in this investigation were made with the same lens; the calibration for that lens is as follows: 1 filar unit = 0.1633μ.

The samples to be tested were mounted in bakelite and polished on SiC abrasive paper. Once a sample had been indented, Knoop hardness was calculated from the length of the long diagonal of the indentation via the following equation:

$$KHN = L/\ell^2 C_p$$

where

KHN = Knoop hardness number, Kg/mm^2

L = Load applied to indentor, Kg

ℓ = Length of long diagonal, mm

C_p = Constant for each indentor
C_p is supplied along with the indentor by the manufacturer. For the FIRL indentor, C_p = 7.028×10^{-2}

The microhardness data for pressure calcintered alumina and spinel are given in Table 9. Typical Optimum Values listed by Lynch, *et al*[12] are as follows: 2000-3000 Kg/mm^2 for alumina; 1200 Kg/mm^2 for spinel. The hardness of the FIRL materials differ somewhat from the

Table 9. MICROHARDNESS DATA

Material	Al_2O_3	Al_2O_3	Al_2O_3	$MgAl_2O_4$	$MgAl_2O_4$	$MgAl_2O_4$
Sample Number	766	774	778	1023	1009	1006
Length of indentions, filar units	602	511	493.5	604	568	587
	600	563	515.5	646	600	563
	565	544	487	568	621	609
	582.5	578	517	569	571	529
	586	580	514.5	552	638.5	548.5
	496	488	440	610	547	575
	548	517	497	547	584.5	551.5
	627	506	572.5	590	573	585
	542	524	533	527	537.5	536
	544	571	520	628	598	669
Average filar units	569.25	538.2	509.0	584.1	586.55	575.3
Average microns	92.96	87.9	83.12	95.4	95.78	93.88
KHN, kg/mm^2	1645	1837	2065	1564	1551	1613

Average KHN, Al_2O_3 1849
Average KHN, $MgAl_2O_4$ 1576

typical values; the spinel is harder and the alumina softer. Additional research will be required to resolve these differences.

The limited microhardness data for nichrome reinforced alumina (Table 10) indicate only moderate degradation of hardness near the wire. The reduced hardness near the wire suggests that wire mesh on the face of a ballistic sample may be undesirable. This possibility will be investigated in Part II of this research, when wire reinforced composites with and without wire on the faces will be ballistically tested.

Table 10. MICROHARDNESS DATA FOR A NICHROME-ALUMINA COMPOSITE

SAMPLE 1034

Distance from wire, m.m.	Length of Indention, Filar Units	Average Length of Indention, microns	KHN, Kg/mm^2
0.2	597 558.5	94.4	1597
0.4	576 588.5	95.1	1577
0.6	603 583.5	95.2	1571
0.8	583 561.5	93.4	1631
1.0	516 500	83.0	2065

SECTION VI

CONCLUSIONS

1. Titania doped Al_2O_3 and $MgAl_2O_4$ ceramics can be fabricated into fully dense, fine grained (1-5μ) polycrystalline shapes at moderate temperature, 1350°C, and pressure, 5000 psi, by the method of pressure calcintering.

2. Extremely high elastic moduli, 66×10^6 psi for Al_2O_3, 47×10^6 psi for $MgAl_2O_4$, can be achieved in Al_2O_3 and $MgAl_2O_4$ ceramics fabricated by pressure calcintering.

3. Nichrome wire mesh can be incorporated into pressure calcintered Al_2O_3 and $MgAl_2O_4$ matrices to form dense composites with little or no degrading of either the wire mesh or the ceramic matrix.

4. Nichrome reinforced Al_2O_3 and $MgAl_2O_4$ composites can absorb significant quantities of energy in fracture after they have initially cracked.

5. Single crystal sapphire whiskers and fibers tend to dissolve in the matrix material and/or fracture and recrystallize during the fabrication of sapphire reinforced Al_2O_3 and $MgAl_2O_4$ composites by pressure calcintering.

6. The inclusion of 0.01" diameter sapphire single crystal fibers into dense, fine grained $MgAl_2O_4$ ceramics results in an increase in the diametral compression tensile strength of the spinel.

SECTION VII

FUTURE WORK

The primary objective of Part II of this research will be to develop optimum materials and configurations for ballistic armor. With this objective in mind the investigators feel that it is important to submit samples for ballistic testing at the earliest possible time, and to use the feedback from those tests to continue optimization.

Four inch diameter disks of nichrome reinforced alumina and spinel with differing volume fractions and spatial arrangements of nichrome wire mesh disks will be fabricated and submitted to the sponsor. The results of ballistic tests on those samples will be used as a guide in selecting optimum composite configurations.

The feasibility of incorporating inconel honeycomb shapes into alumina and spinel matrices will be investigated with small, 0.75" diameter, samples. If the honeycomb reinforcement looks promising, 4" diameter cylinders will be fabricated for ballistic tests.

Of the sapphire reinforced composites fabricated in this investigation, only 0.01" diameter sapphire fiber reinforced spinel shows any promise. Further fabrication and testing of 0.75" diameter samples will be carried out to ascertain the desirability of making ballistic test samples.

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13 ABSTRACT

Dense ($\geq$ 99%), fine grained ($\leq$ 1-5) samples of titania doped Al_2O_3 and $MgAl_2O_4$ have been fabricated by pressure calcintering. Extremely high values of Young's modulus have been realized in these materials; average values were 66 x 10^6 psi for Al_2O_3, 47 x 10^6 for spinel. Composites were formed by the addition of nichrome wire mesh, single crystal sapphire fiber, and sapphire whiskers. Microstructural examination shows little or no degradation of the wire reinforcement, but the sapphire fibers and whiskers recrystallize and tend to dissolve in the ceramic matrices. Diametral compression tensile strengths are lowered by the inclusion of nichrome wire, but the resultant composites absorb significant amounts of energy during testing after the initial crack. Mechanical properties of sapphire-Al_2O_3/$MgAl_2O_4$ composites were unimpressive with the exception of single crystal fiber - $MgAl_2O_4$ composites. The inclusion of single crystal sapphire fibers in the spinel matrix resulted in a higher ultimate tensile strength than that of the unreinforced spinel ceramic.

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Alumina - Nichrome						
Alumina - Sapphire						
Spinel - Nichrome						
Spinel - Sapphire						
Ceramic Armor						

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